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## (54) THE UTILISATION OF PAPER SLUDGE FOR SHEETING OR MOULDING

(71) We, NATIONAL RESEARCH DEVELOPMENT CORPORATION, a British corporation established under statute, of Kingsgate House, 66/74 Victoria Street, London, S.W.1E 6SL, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

The present invention relates to the utilisation of "paper sludge" for sheeting or moulding.

The waste effluent obtained in paper manufacture is a dilute aqueous sludge containing vegetable fibres which are short enough to pass through the wires of the Fourdrinier machine and inorganic material such as titanium oxide and clays. The sludge presents a serious disposal problem in paper manufacture. Dumping of this heavy and bulky material is expensive in view of its water content (approximately 60 to 95% by weight water) and, after dumping, the decay of the fibrous material in the sludge produces an obnoxious residue. Furthermore disposal sites are limited and have to be agreed upon with local authorities.

The invention provides a method of recovering the fibrous and mineral material in "paper sludge" (as herein defined) in which textile and/or mineral fibres with an average fibre length of from 1/16" to 5" are added to the slurry as individual fibres, or as fragments of woven or like fabric so as to assist in agglomerating together the fibre material of the slurry together with at least part of any mineral content thereof, and in which the agglomerated material is recovered as a board product or as a moulding composition.

The invention also provides a moulding composition comprising an agglomerated

mixture of dehydrated or partially dehydrated "paper sludge" (as herein defined) together with added textile and/or mineral 45 fibres with an average fibre length of from 1/16th of an inch to 5 inches.

The invention further provides a process for the production of a moulded article which comprises depositing onto a liquid 50 permeable support to form an agglomerated layer a slurry containing cellulose fibres and inorganic minerals with an average fibre length of 1/16" to 5" and also vegetable fibres of below 1/16" in average fibre length 55 which originate from effluent obtained in paper manufacture, passing the layer thus formed between pressure rollers to squeeze out part of its water content and moulding the sheet under pressure to form the desired 60 article.

By "paper sludge" we mean a dilute aqueous slurry or sludge originating as a waste effluent from paper manufacture, the slurry or sludge containing vegetable fibres 65 of below 1/16th inch in average fibre length.

Mineral fibres such as rock wool, asbestos or the like may be used as the added fibres, and a board product according to the invention may be laminated or moulded onto 70 a mineral fibre batten or formed with an apertured surface or laminated to a perforated metal or plastics sheeting or wire mesh.

Substantial additions of mineral fibres increase the fire resistance of boards and 75 mouldings, e.g. for use as ascoustic boards and partitions.

The board may constitute a core or a surface lamination of a fire resistant partition and/or insulation panel. It may also be 80 used as a fire resistant edging or moulding.

In one form of the invention, the mixture is deposited as a layer of agglomerated fibrous material onto a liquid-permeable

support and the layer thus formed is passed between pressure rollers to squeeze out water with added suction if required so as to form a sheet.

5 The method of the invention is particularly applicable to the treatment of aqueous slurries with very short fibres obtained at the Fourdrinier stage but can be used to recover longer fibres from waste slurries  
10 obtained in paper manufacture. The invention is based on the discovery that by mixing with the waste slurry further fibrous material with an average fibre length greater than that of the fibrous material escaping  
15 from the paper-making process, the shorter fibres in the slurry can be matted together with the added fibres into the form of a loose sheet which is readily recoverable from the slurry.

20 We have surprisingly found that even non-felting fibres such as synthetic fibres assist in matting together the original fibres.

The method can be carried out using as starting materials slurries containing as little  
25 as 10% or less by weight of fibrous material, and even as little as 1% by weight. The waste slurry usually contains inorganic minerals such as titanium oxides, silica, pigment residues and clays which are  
30 of value in the final product and help in the matting step. The fibrous materials which is added may comprise waste non-felting synthetic fibres such as waste glass, nylon, Terylene (trade mark) or polypropylene  
35 fibres or monofilaments. Other synthetic fibres may also be used or mixtures of waste non-felting synthetic fibres or plastics impregnated or coated material fibres with waste felting fibres such as wood fibres or  
40 wool, cotton or rag waste. Textile wastes containing mixed natural and synthetic fibres which are at present unusable because of the high cost of separation into the fibres of different nature may usefully  
45 be employed.

The fibrous material mixed with the slurry to produce matting is preferably added in the dry state. It may be pretreated, e.g. by  
50 charging the fibres electrically or coating them with an adhesive, so as to promote the adherence thereto of the shorter fibres already in the slurry.

The added fibrous material may also be pretreated with cationically surface-active  
55 agents, flocculating agents for promoting agglomeration in the slurry, bactericidal agents, disinfectants, pigments or colourants, and any of these materials can also be added to the liquid of the slurry before or  
60 after the supply of additional fibres.

The product of the invention may be usable as such as a moulding composition if its mineral content is sufficiently high when the required amount of water has been  
65 eliminated, e.g. to yield a product with less

than 10% by weight of water. Synthetic resins such as phenolic or ureaformaldehyde resins may be added to produce the required moulding properties.

The fibrous material from the mixture is  
70 preferably deposited on a perforated travelling band which may be similar, for example, to a fine mesh Fourdrinier screen with or without a suction base. If the mixture run onto the wire screen contains less  
75 than 10% by weight of fibrous material, then the content of fibrous material is preferably brought up to approximately 10% by weight by passing the mixture over a  
80 filter such as a rotating perforated drum, scraping the thickened mixture off the drum and delivering it to the screen. The drum may contain a layer of a porous blanket or foamed material which allows water to pass  
85 through but prevents the passage of solids, thus allowing only clean water to go to waste.

When using dilute slurries of fibrous material (less than 10% content of fibrous material), the additional fibrous material can  
90 be added before or after the slurry is thickened, e.g. by passage over a perforated drum. An advantage of adding the fibrous material to the very dilute slurry before thickening to a fibrous solids content  
95 of 10% by weight or more is that good distribution and mixing of the additional fibres are more easily achieved in the presence of a large excess of water. The "dried sludge" product is very much less bulky  
100 than the original sludge and a considerable reduction in total weight is achieved. Furthermore, an unsaleable material disposable only with difficulty and at high cost may be transformed after moulding into saleable  
105 mouldings, e.g. flues, roofing parts, corrugated mouldings or box covers.

One example of a method according to the present invention is now described by way  
110 of example.

The method relates to the production of sheet products from the waste effluent of a paper-board mill. In this case, the effluent contains short vegetable fibres in an amount  
115 of 1% or less by weight. Other solid material is, however, normally present in the effluent in an amount commonly up to 1% by weight or more giving a total solids content of up to 2% by weight or slightly more. The non-fibrous, solid materials are mineral  
120 dust, for example, titanium dioxide and clay, and may sometimes constitute 75% by weight of the dry solid content of the waste effluent.

A quantity of dry fibrous material is  
125 added in an amount of from 10% to 30% by weight based on the total dry weight of the solid material in the effluent or in an amount of 100% of the dry weight of fibrous material only in the slurry.  
130

The fibrous material which is added may be, for example, waste chopped rags, sisal, jute, hessian, nylon, polypropylene choir, terylene, wool, asbestos, glass fibre, rubberised fibres from old tyres or metallic fibres, and also wide mesh or netting or very porous fabrics can be used to accumulate the solid material of the sludge. Chopped waste rags and sisal or jute waste dust from weaving sheds are advantageous materials to use from the point of view of economy.

When mixed with the effluent, the dry fibres of the additional fibrous material pick up the fibres in the effluent and form therewith agglomerations of fibres. The agglomeration step is helped by the fact that the additional fibrous material (average fibre length from 1/16" to 5", preferably from 1/8" to 3") as a greater average fibre length than the fibrous material in the effluent. The agglomerated material is easier to filter than the elusive solid content of the original waste sludge.

When the additional fibrous material has been mixed with the effluent and/or the effluent been deposited thereon, the resulting mixture is thickened to a higher solids content. The thickening can be effected in a number of ways. The mixture, which normally contains a total of less than 2% by weight of fibrous solid material and less than 3% by weight of solid material, is run onto a rapidly rotating perforated drum containing a layer of a porous blanket or foamed material, which absorbs the water but retains the solids. The material is held on the outer surface of the perforator drum mainly because the added fibres form a layer which is scraped off by a fixed doctor blade and is delivered onto the upper surfaces of a perforated travelling band, constituting a liquid-permeable support, mounted like an endless conveyor with its upper surface horizontal or slightly inclined to the horizontal.

The layer of material in dough-like form coming off the travelling band continuously and containing not more than 10% by weight of total solids (fibrous and non-fibrous) is passed through a series of heavy rubber-covered pressure rollers in which water is squeezed out to bring the total solids content up to approximately 30% by weight. The material emerging from the rollers can be cut to form sheets which are stacked and allowed to dry out of doors. Preferably, before being stored for drying, the stacks of sheets are subjected to pressure for a period of time, advantageously several days, e.g. three or four days, by which time they have gained appreciably in strength.

Alternatively, the sheets from the rollers can be stacked and the stacks subjected to pressure between plates whilst the sheets are dried by heating or by means of warm

air. Alternatively, drying agents such as dehydrated lime may be used to remove residual water.

The sheets or mats which are thus produced can serve as "fillers" in manufacturing board products, for example as cores for building boards or light insulation boards, or reinforced plastics sheets or moulded articles.

The sheets can serve as satisfactory products for some purposes without the addition of any binding material since the process of depositing the fibres onto a filter band and subsequent rolling brings about mechanical interlocking of the fibres which contributes to the strength of the final sheet. A further result of the rolling and squeezing to which the fibrous material is subjected is to squeeze out the non-fibrous mineral particles which form a high proportion of the original solids content of the effluent. The mineral dust is squeezed out of the fibrous material to a large extent together with the water and the proportion of non-fibrous mineral in the sheet material is thus substantially reduced.

If sheets having a greater strength are required, some cement, gypsum, plaster or dehydrated lime can be added during or after the rolling procedure described above. Such additions assist in removing water by taking it up chemically as well as by increasing the strength of the board in providing a binding action. Synthetic plastics materials, for example, phenolic and urea-formaldehyde resins to an amount of 2% to 3% by weight based on the weight of the dry sheet can also be added as binders and these and the chemicals referred to above can be added by methods such as spraying, coating or impregnation. The synthetic plastics materials which can be used include acrylic emulsions and polyvinyl acetate emulsions. The plastics materials can be added as well as, or instead of, the other binding materials mentioned above. The fibrous material can be impregnated or treated with chemicals such as aluminium sulphate to promote better adhesion to cement or other mineral binders.

The above description has referred to the pressing of stacks of sheets but the sheets can be pressed individually or in small numbers of two or three, for example, if desired. If a cement is added, the amount of water in the sheets to be pressed, that is the sheet material leaving the rollers which squeeze out the water, should advantageously be just sufficient to react with the cement which has been added. In any case, whether or not cement is added, it has been found advantageous for the water content of the sheets to be such that water just begins to seep out of the sheets as the final pressing is carried out in the press.

The method described above is concerned with processing waste effluent in the form in which it comes from a Fourdrinier machine containing from  $\frac{1}{2}\%$  to 2% by weight of total solids. However, the method can be applied to processing the effluent after it has been allowed to thicken by standing in filter beds and when it contains 10% by weight of total solids. Additional fibrous material is mixed with the effluent in the same range of proportions as described above and the same steps are carried out.

Although the invention can be applied to batch processing, it is readily carried out as a continuous method. In a continuous method of production, the mixing in of the additional fibrous material can be carried out in a tank through which the dilute fibrous slurry flows, the mixture emerging from the tank being supplied to a thickening apparatus, to increase the solids content, or direct to the perforated travelling band. If additives such as cement are added, they can be applied to the continuously moving layer or sheet before or during rolling.

The lengths of the moving sheet material can be cut off automatically to form the sheets which can be mechanically fed directly to the pressing mould, thus making a completely automatic process.

Laminated products can be prepared from the sheets produced by the method described above. For example, attractive board products can be produced by moulding under pressure and with heating a board according to the invention containing an amino or phenolic resin and a melamine sheet which are thereby laminated together.

The product of the invention may be used as a dough moulding composition after the water has been eliminated to a large extent, i.e. the moisture content has gone down to below 10%. If desired, synthetic materials such as urea-formaldehyde resins and phenolic resins may be added. These dough moulding compositions can be moulded into a variety of shapes which can be used in the building industry or in the packaging industry. They are preferably moulded with the use of heat and pressure.

There is always a considerable mineral content in the original sludge which is deposited as fine particles onto the added fibres in the product of the invention. When mixed with cement or like material, the product resembles asbestos cement products in being fire-resistant and the expense of adding fire retarding agents may be avoided.

Very hard and dense materials can be made if pressure of at least from  $1/4$  ton to 3 tons per square inch is used together with synthetic resins such as phenolic resins and these materials have a similar appearance to laminated and impregnated wood, with lower pressures, insulating boards are

materials of panels may be obtained.

Various means may be used to help in the elimination of water, e.g. slurry pumps in which water is pressed through a filter (Mono (trade mark) pumps).

The invention is now illustrated by the following specific examples of the production of boards according to the invention. In all cases, the added textile and/or mineral fibres had an average fibre length of from  $75 \frac{1}{16}''$  to  $5''$ .

#### Example 1

40 gm of waste chopped rags are mixed into 2 kg of sludge from a paper mill (10% by weight total solids content) using an internal dough mixer giving an even distribution of the constituents of the mixture. The mixture is layered onto cotton duck belting and passed through a tight nip of heavy rubber-covered nip rollers to produce a compressed damp board having a total solids content of 33% by weight. This board when dry has appreciable strength.

#### Example 2

40 gm of dry waste chopped rags and 200 gm Canadian asbestos are mixed with 2 kg of paper mill sludge (10% by weight total solids content). The resulting mass is layered and mangled through the nip of heavy rubber-covered rollers to squeeze out some of the water. 500 gm of plaster are then added and the mass is charged into a mould to set to shape.

#### Example 3

0.33 lb of dry waste mixed synthetic fibres is mixed into 10 lbs. of paper mill sludge (10% by weight of total solids content). The mixture is layered and mangled. 8 ozs. of cement and 45 gm of a polyvinyl acetate emulsion (P.V.A. Vandyke 7085) are then added and the mass is moulded to form a building board which has useful sound absorption and heat insulating properties.

The present invention includes sheet products made by the method described above. The sheet products may be used when still moist and soft as semi-manufactured products in the manufacture of finished boards and the invention includes sheet products in the moist soft state.

Further examples are now given:

#### Example 4

A cement board was made from the following materials.

	Batch Weight	Dry Weight	
12% Waste paper slurry	2000 gms.	240 gms.	120
Waste rag fibres	80 gms.		
Cement	300 gms.		125
P.V.A. (Cinapas) 6100	100 gms.		
Calcium Oxide	100 gms.		
Water and Cementone (trademark) No. 8 Hardener in a 4:1 ratio by volume	150 ccs.		130

The above materials were mixed together in a dough mixer to the consistency of a stiff paste. The mixture was layered onto blockboard between two parallel wooden runners 7/8" thickness. The layering was done to a thickness of approximately 4" and the layer was covered with a sheet of polythene (to prevent sticking) and then rolled with a steel roller. Rolling was continued until the material was compressed to 7/8" thick (the 7/8" wooden runners acting as stops). A wet consolidated board was thus obtained.

This board was allowed to dry naturally for 48 hours and finally gently oven dried to hasten the process.

A compact rigid board was obtained.

#### Example 5

Example 4 was repeated with the following modifications.

The 2 kg of 12% paper mill sludge (240 gms dry) and the 80 gms of waste rag fibres were mixed in a dough mixer with 100 gms of aluminium sulphate. This mixture was left overnight in an airtight container to allow a mineralising action to take place.

The following day the mixture was re-charged into the dough mixer and the following constituent were added thereto.

Cement	300 gms.
P.V.A. (Vinapas 6100)	100 gms.
Calcium oxide	100 gms.
Water and Cementone No. 8	
Hardener in a 4:1 ratio by volume	150 ccs.

The manufacture of a board was then carried out as described in Example 4.

#### Example 6

A 6' 0" x 2' 0" cement board was produced utilising sludge from a paper mill.

Formulation	Batch Weight
12% Waste paper slurry	50 lbs
Waste rag fibres	(5½ lbs, dry weight)
Cement	2 lbs.
P.V.A. (Vandyke 7085)	8 lbs.
Calcium oxide	10.5 lbs.
Water 4 parts	2 lbs.
Cementone No. 8 1 part	500 ccs.

This formulation was mixed in an internal dough mixer until a stiff paste consistency was obtained. The mixture was then layered onto a large sheet of Blockboard (trade mark) to which two 7/8" thick parallel wooden runners had been fastened. The paste was consolidated by rolling with a steel roller as in Examples 4 and 5 until a 7/8" thick 6' 0" x 2' 0" wet board was obtained. A ribbed pattern was embossed on one-half of the board by passing a wire

bound wooden roller over it.

Finally a piece of Blockboard was placed on the wet top surface of the product and nailed down to create a lock mould. The board was then left to mature.

#### Example 7

A plaster based board was produced utilising sludge from a paper mill.

Formulation	Parts	Dry	Batch Weight	75
12% Waste paper slurry	50	5½	2000 gms.	
Waste rag fibres	2	2	40 gms.	
Thistle (trade mark) plaster			500 gms.	80
Carey Canadian Asbestos			200 gms.	

The above formulation was mixed with a little water to form a stiff paste and subsequently rolled out into a board as in Examples 4 to 6. The wet board was embossed with a pattern on one surface by pressing a sheet of embossed aluminium against it. The embossed board was oven dried and finally painted with polyvinyl acetate.

#### Example 8

A wood-like material was produced with phenolic resin, using preformed material obtained from waste paper sludge.

Formulation	
12% sludge from a paper mill	50 lbs. WET
Waste rag fibres	2 lbs.

The above was mixed in an internal dough mixer until a stiff paste consistency.

Ten pounds weight of this paste were layered between two pieces of cotton duck to form a layer 12" wide, 52" long and 3/8" thick. The layer was passed through a pair of rubber-covered steel nip rollers set at minimal pressure. After squeezing, the weight was 4 lbs. 15 ozs. The whole was then passed once more through the nip rollers with an extra nip applied.

On re-weighing, the weight of the squeezed biscuit material was found to be 5 lbs. 4 ozs.

#### Impregnation

The impregnating resin solution was as follows:

C1151/76 Sternite (trade mark)	
Phenolic Resin (76% solids)	3 gallons
Water	1½ gallons

Six 9" x 9" squares were cut from the biscuit material which had been dried in a hot air oven and these were impregnated in the resin solution for 4 hours. After immersion, the 9" square biscuits were dried for a period of 4½ hours at 180°C.

#### Moulding

Five of these resin-impregnated biscuits were then placed one on top of the other in a 9" square mould, and pressed at 150°C. for ten minutes.

The product was a well laminated board of wood-like appearance and of a very tough nature.

**WHAT WE CLAIM IS:**

1. A method of recovering the fibrous and mineral material in "paper sludge" (as herein defined) in which textile and/or mineral fibres with an average fibre length of from 1/16" to 5" are added to the slurry as individual fibres, or as fragments of woven or like fabric so as to assist in agglomerating together the fibre material of the slurry together with at least part of any mineral content thereof, and in which the agglomerated material is recovered as a board product or as a moulding composition.
2. A method according to claim 1, wherein the mixture obtained by the addition of the textile and/or mineral fibres is deposited as a layer of agglomerated fibrous material onto a liquid-permeable support, and the layer thus formed is passed between pressure rollers to squeeze out part of its water content and the resulting sheet is dried or moulded or impregnated.
3. A method according to claim 1 in which a synthetic resin material and/or a filler is added to the mixture obtained by the addition of the textile fibres.
4. A method according to any of claims 1 to 3 in which cement, gypsum or aluminium sulphate is added to the mixture obtained by the addition of the textile fibres.
5. A method according to claim 1 substantially as herein described with reference to any of the specific examples.
6. A board product or a moulding composition when produced by a method as claimed in any of claims 1 to 5.
7. A board product according to claim 6 in which mineral fibres such as rock wool, asbestos or the like are used as the added fibres, and the board product is laminated or moulded onto a mineral fibre batten or

formed with an apertured surface or laminated to a perforated metal or plastics sheeting or wire mesh.

8. A fire resistant partition and/or insulation panel comprising a core or a surface lamination constituted by a product as claimed in claim 6.
  9. A fire-resistant edging or moulding comprising a product as claimed in claim 6.
  10. A moulding composition comprising an agglomerated mixture of dehydrated or partially dehydrated "paper sludge" (as herein defined) together with added textile and/or mineral fibres with an average fibre length of from 1/16th of an inch to 5 inches.
  11. A process for the production of a moulded article which comprises depositing onto a liquid permeable support to form an agglomerated layer a slurry containing cellulose fibres and inorganic minerals with an average fibre length of 1/16" to 5" and also vegetable fibres of below 1/16" in average fibre length which originate from effluent obtained in paper manufacture, passing the layer thus formed between pressure rollers to squeeze out part of its water content and moulding the sheet under pressure to form the desired article.
  12. A moulding composition as claimed in claim 10 substantially as herein described with reference to any of the specific examples.
  13. A process for the production of a moulded article as claimed in claim 11, substantially as herein described with reference to any of the specific examples.
  14. A moulded article when prepared by a process as claimed in claim 11 or 13.
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